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IS 6503 (1988): Method for quantitative chemical analysis of ternary mixtures of protein fibres, nylon 6 or nylon 6.6 and certain other fibres [TXD 5: Chemical Methods of Test]



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भारतीय मानक

प्रोटीन रेशों, नायलोन 6 अथवा नायलोन 6.6 और कुछ अन्य
रेशों के त्रयंगी मिश्रण के परिमाणात्मक रासायनिक विश्लेषण
की भारतीय मानक पद्धति
(पहला पुनरीक्षण)

Indian Standard

METHODS FOR QUANTITATIVE CHEMICAL ANALYSIS OF
TERNARY MIXTURES OF PROTEIN FIBRES, NYLON 6 OR
NYLON 6.6 AND CERTAIN OTHER FIBRES

(First Revision)

First Reprint AUGUST 2005)

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 30 September 1988, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

This Indian Standard was published in 1972 and has been revised to include another method based on the dissolution of polyamide in 18 percent (*m/m*) (5.36 N) (sp gr at 20°C = 1.0878) hydrochloric acid, developed by the Textiles Committee, Bombay. In addition, use of sodium hydroxide or potassium hydroxide has been specified for dissolving protein fibres in place of sodium hypochlorite because of difficulty in preparation of sodium hypochlorite solution and its less stability in respect of available chlorine during storage.

The use of different fibre blends in textiles has necessitated the formulation of standard methods for identification and quantitative estimation of respective fibres. The quantitative analysis of textile fibres in mixtures is of considerable importance to the textile technologists, traders and consumers.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

AMENDMENT NO. 1 FEBRUARY 1992
TO
IS 6503 : 1988 METHODS FOR QUANTITATIVE
CHEMICAL ANALYSIS OF TERNARY MIXTURES OF
PROTEIN FIBRES, NYLON 6 OR NYLON 6.6 FIBRES
AND CERTAIN OTHER FIBRES

(First Revision)

(This amendment is being issued to remove any ambiguity regarding values of commercial moisture regain of different fibres to be taken into consideration at the time of calculating the test results.)

(*Page 3, clause 7.3.3, Note 1*) — Substitute the following for the existing Note :

“For the purpose of calculations the commercial moisture regain values for various fibres as specified in IS 13157 : 1991 ‘Textiles fibres — Commercial moisture regains — Specification’ shall be used.”

(*Page 3, clause 7.3.3, Note 2*) — Delete.

(TXD 5)

Indian Standard

METHODS FOR QUANTITATIVE CHEMICAL ANALYSIS OF TERNARY MIXTURES OF PROTEIN FIBRES, NYLON 6 OR NYLON 6.6 AND CERTAIN OTHER FIBRES

(First Revision)

1 SCOPE

This standard (First Revision) prescribes two methods for the quantitative chemical analysis of ternary mixtures, after removal of non-fibrous matter, of natural or regenerated protein fibres and nylon 6 or nylon 6.6, and any of the following fibres:

- a) cotton or viscose rayon (including polynosic fibre),
- b) glass, or
- c) polyester fibre.

NOTE — Before conducting an analysis according to this standard, the fibres present in the mixture should be identified (*see* IS 667 : 1981) and the sample to be analysed should be freed from all non-fibrous matter (*see* IS 9068 : 1979). Dye in the dyed fibres is considered to be an integral part of the fibre and is not to be removed.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
IS 667 : 1981	Methods for identification of textile fibres (<i>first revision</i>)
IS 1070 : 1977	Specification for water for general laboratory use (<i>second revision</i>)
IS 9068 : 1979	Recommended methods for the removal of non-fibrous matter prior to quantitative analysis of fibre mixtures

3 SAMPLING

3.1 Lot

The quantity of textile material of one definite type and quality delivered to a buyer against one despatch-note shall constitute a lot.

3.1.1 If the textile material is fibre or yarn and the lot consists of more than 200 kg of fibre or yarn, it shall be divided into sub-lots, each weighing 200 kg or less.

3.1.2 Each sub-lot shall be tested separately.

3.2 Sampling for Fibre and Yarn

From a sub-lot, 15 increments, each approximately weighing 10 g, shall be taken from different parts and mixed thoroughly. This shall constitute a test sample.

3.3 Sampling for Fabrics

3.3.1 The number of pieces to be selected shall be in accordance with Table 1. The pieces thus selected shall constitute a gross sample.

Table 1 Sample Size

(Clause 3.3.1)

Lot Size (Number of Pieces)	Sample Size (Number of Pieces)
Up to 100	3
101 to 300	4
301 to 500	5
501 and above	7

3.3.2 From each piece in the gross sample selected as in 3.3.1 cut out small portions from at least two different parts weighing about 25 g. The parts selected shall represent the gross sample as far as possible. In the case of fabrics with a definite repetition in weave pattern, the parts selected shall include all yarns in the complete repeat. Dissect the small portions of fabric thus collected into yarn and mix them thoroughly.

4 APPARATUS

4.1 Sintered Glass Filter Crucible

Of appropriate capacity with pore size of 90 to 150 microns (porosity 1) and provided with a ground glass stopper. If stopper is not available, the crucible should be enclosed in weighing bottle for weighing.

4.2 Desiccator

Charged with self-indicating silica gel or anhydrous calcium chloride.

4.3 Ventilated Oven

For drying samples at $105 \pm 3^\circ\text{C}$.

4.4 Analytical Balance

Capable of weighing to an accuracy of 0.0002 g.

4.5 Conical Flask

Of 100 ml minimum capacity, provided with a ground glass stopper.

4.6 Filter Flask

With connection to filter pump and adaptor to enable the crucible (4.1) to be fitted to it.

4.7 Mechanical Shaker

5 REAGENTS

5.0 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (see IS 1070 : 1977) shall be used where the use of water as a reagent is intended.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the test results.

5.1 Alkali Solution

5.1.1 Potassium Hydroxide Solution

5 percent (*m/m*).

or

5.1.2 Sodium Hydroxide Solution

5 percent (*m/m*).

5.2 Acetic Acid

Dilute solution, 5 percent (*m/m*).

5.3 Formic Acid

80 percent (*m/m*) of sp gr 1.186 at 20°C . Dilute 880 ml of 90 percent (*m/m*) of formic acid (sp gr 1.204) to one litre with water. Alternatively, dilute 780 ml of 98 to 100 percent formic acid (*m/m*) (sp gr 1.22) to one litre with water. The concentration is not critical within the range 77 to 83 percent (*m/m*) (sp gr 1.181 to 1.191) formic acid.

5.4 Ammonia

Dilute solution, 80 ml of concentrated ammonia solution (sp gr 0.89) diluted to one litre with water.

5.5 Hydrochloric Acid Solution

18 percent (*m/m*) (5.36 N) (sp gr at 20°C = 1.0878).

6 PREPARATION OF TEST SPECIMENS

From the sample, after removing size and finishes as recommended in IS 9068 : 1979, draw a representative sample weighing about 2 to 3 g. Cut the

yarn into pieces and dissect the cloth into yarn pieces of about 10 mm length.

7 METHOD 1

7.1 Principle

A sample of the mixture is dried and weighed. The protein fibre is dissolved in sodium hydroxide or potassium hydroxide solution of a given strength. The residue is collected, washed, dried and weighed. The nylon 6 or nylon 6.6 fibre is then dissolved out from the residue with aqueous formic acid and the insoluble fibre (third component) collected, washed, dried and weighed, its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentage of protein and nylon 6 or nylon 6.6 fibres are found by difference. Alternatively, the nylon 6 or nylon 6.6 is first removed from the mixture with formic acid and the protein fibre then dissolved out from the residue with sodium or potassium hydroxide. The component (protein, nylon 6 or nylon 6.6 fibre) present in the larger quantity should be dissolved first.

7.2 Procedure

7.2.1 Take from the pre-treated sample (see 6.1) a test specimen weighing about 1 g. Dry it in a weighing bottle at $105 \pm 3^\circ\text{C}$ to constant mass, cool in a desiccator and weigh it to obtain the oven-dry mass of the specimen.

NOTE — The mass shall be taken as constant if the difference between any two successive weighings at an interval of 20 minutes does not exceed 0.1 percent.

7.2.2 Transfer the specimen to a 250 ml glass beaker, add 100 ml of sodium or potassium hydroxide solution (previously boiled to expel air) per gram of the specimen. Boil for 10 minutes. Filter the contents of the beaker through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the beaker with a little more sodium or potassium hydroxide solution. Drain the crucible with suction and wash the residue on the filter successively with water, dilute acetic acid and finally water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity). Finally, drain the crucible with suction, dry the crucible and residue at $105 \pm 3^\circ\text{C}$ to constant mass, cool in a desiccator and weigh them.

7.2.3 Transfer the residue by means of forceps to the conical flask, add 100 ml of formic acid (see 5.3) per gram of specimen, insert the stopper, shake the flask to wet out the specimen and allow the flask to stand for 15 minutes at room temperature shaking it at intervals. Filter the contents of the flask through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a little more formic acid. Drain the crucible with suction and wash the residue on the filter successively with formic acid, hot water, dilute ammonia solution,

and finally cold water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity.) Finally, drain the crucible with suction, dry the crucible and residue at $105 \pm 3^\circ\text{C}$ to constant mass, cool in a desiccator and weigh them.

NOTE — The component (protein, nylon 6, or nylon 6.6) present in larger quantity should be dissolved first.

7.2.4 Repeat the procedure prescribed in 7.2.1 to 7.2.3 with the remaining test specimen(s).

7.2.5 Alternatively, using the procedure described above, first dissolve the nylon with formic acid and subsequently dissolve the protein with sodium or potassium hydroxide solution.

7.3 Calculations

7.3.1 Calculate the mass of each component as a percentage of the total mass of the fibres in the mixture by any of the methods as prescribed in 7.3.2, 7.3.3 or 7.3.4.

7.3.2 Method Based on Clean Dry Mass

Calculate the percentages of clean dry component by the formula:

$$P_1 = 100 - (P_2 + P_3)$$

$$P_2 = \frac{100 d_1 \left[r_1 - \frac{d_2}{d_3} \times r_2 \right]}{m}$$

$$P_3 = \frac{100 d_2 \times r_2}{m}$$

where

P_1 = percentage of clean dry component 1 (first soluble) component (see 7.2.2);

P_2 = percentage of clean dry component 2 (second soluble component) (see 7.2.3);

P_3 = percentage of clean dry component 3 (insoluble component);

m = dry mass of the specimen;

r_1 = dry mass of the residue after removing component 1 with first reagent;

r_2 = dry mass of the residue after removing components 1 and 2 with first and second reagents;

d_1 = correction factor for loss in mass of component 2 in first reagent (see Note);

d_2 = correction factor for loss in mass of component 3 in first reagent (see Note); and

d_3 = correction factor for loss in mass of component 3 in first and second reagents.

NOTE — The value of correction factors d_1 , d_2 and d_3 depending upon the nature of component fibres are given below:

Fibre	Sodium Hydroxide	Potassium Hydroxide
Cotton	1.06	1.05
Viscose rayon	1.05	1.04
Polyester	1.04	1.02
Other fibres	1.00	1.00
Polyamide	1.01	1.00

7.3.3 Method Based on Clean Dry Mass with Percentage Addition of Agreed Value for Moisture Regain

Calculate the percentages of clean dry component with additions for moisture, by the formula:

$$P_{1M} = \frac{100 P_1 \left[1 + \frac{a_1}{100} \right]}{P_1 \left[1 + \frac{a_1}{100} \right] + P_2 \left[1 + \frac{a_2}{100} \right] + P_3 \left[1 + \frac{a_3}{100} \right]}$$

$$P_{2M} = \frac{100 P_2 \left[1 + \frac{a_2}{100} \right]}{P_1 \left[1 + \frac{a_1}{100} \right] + P_2 \left[1 + \frac{a_2}{100} \right] + P_3 \left[1 + \frac{a_3}{100} \right]}$$

$$P_{3M} = \frac{100 P_3 \left[1 + \frac{a_3}{100} \right]}{P_1 \left[1 + \frac{a_1}{100} \right] + P_2 \left[1 + \frac{a_2}{100} \right] + P_3 \left[1 + \frac{a_3}{100} \right]}$$

where

P_{1M} = percentage of clean dry component 1 with percentage additions for moisture,

P_{2M} = percentage of clean dry component 2 with percentage additions for moisture,

P_{3M} = percentage of clean dry component 3 with percentage additions for moisture,

a_1 = percentage addition to component 1 for moisture,

a_2 = percentage addition to component 2 for moisture, and

a_3 = percent addition to component 3 for moisture.

NOTES

1 The following values for standard moisture regain of various fibres may be considered:

Fibre	Standard Moisture Regain (Percent)
Aramid (safety apparel fabrics)	4.5
Cotton	8.5
Nylon (polyamide)	4.5
Polyester	0.4
Rayon (regenerated cellulose)	13.0
Regenerated protein	10.0
Silk	11.0
Textile glass	0
Wool	13.6

2 The standard moisture regain values are generally accepted as the commercial moisture regain values in the trade.

7.3.4 Method Based on Clean Dry Mass with Percentage Additions for Moisture and Non-fibrous Matter

Calculate the percentages of clean dry components with additions of moisture and non-fibrous matter by the following formula (see also Notes 1 and 2 under 7.3.3) :

$$P_{1A} = \frac{100 P_1 \left[1 + \frac{a_1 + b_1}{100} \right]}{P_1 \left[1 + \frac{a_1 + b_1}{100} \right] + P_2 \left[1 + \frac{a_2 + b_2}{100} \right] + P_3 \left[1 + \frac{a_3 + b_3}{100} \right]}$$

$$P_{2A} = \frac{100 P_2 \left[1 + \frac{a_1 + b_1}{100} \right]}{P_1 \left[1 + \frac{a_1 + b_1}{100} \right] + P_2 \left[1 + \frac{a_2 + b_2}{100} \right] + P_3 \left[1 + \frac{a_3 + b_3}{100} \right]}$$

$$P_{3A} = \frac{100 P_3 \left[1 + \frac{a_1 + b_1}{100} \right]}{P_1 \left[1 + \frac{a_1 + b_1}{100} \right] + P_2 \left[1 + \frac{a_2 + b_2}{100} \right] + P_3 \left[1 + \frac{a_3 + b_3}{100} \right]}$$

where

P_{1A} = percentage of pure dry component 1 with percentage additions for moisture and non-fibrous matter,

P_{2A} = percentage of pure dry component 2 with percentage additions for moisture and non-fibrous matter,

P_{3A} = percentage of pure dry component 3 with percentage additions for moisture and non-fibrous matter,

a_1 = percentage addition to component 1 for moisture,

a_2 = percentage addition to component 2 for moisture,

a_3 = percentage addition to component 3 for moisture,

b_1 = percentage addition to component 1 for non-fibrous matter,

b_2 = percentage addition to component 2 for non-fibrous matter, and

b_3 = percentage addition to component 3 for non-fibrous matter.

NOTE — The percentage additions for non-fibrous matter may be as agreed to between the buyer and the seller

8 METHOD 2

8.1 Principle

8.1.1 The protein fibre is dissolved out from a known dry mass of the mixture, with 5 percent sodium hydroxide or potassium hydroxide solution (m/m). The residue is collected, washed, dried and weighed. The nylon 6 or nylon 6.6 fibre is then dissolved out from the residue with 18 percent hydrochloric acid solution (m/m) and the insoluble fibre (third component) is collected, washed, dried and weighed; its mass, corrected if necessary, is expressed as a percentage of the dry mass of the mixture. The percentages of protein and nylon 6 or nylon 6.6 are found by difference. Alternatively, the nylon 6 or nylon 6.6 is first removed from the mixture with hydrochloric acid and the protein fibre then dissolved out from the residue with sodium hydroxide or potassium hydroxide solution. The component (protein, nylon 6 or nylon 6.6 fibre) present in the larger quantity should be dissolved first.

8.2 Procedure

8.2.1 Follow the procedure described in 7.2.1.

8.2.2 Transfer the specimen to a 250 ml glass beaker, add 100 ml of 5 percent (m/m) sodium hydroxide or potassium hydroxide solution (previously boiled to expel air) per gram of specimen, shake the beaker to wet out the specimen and boil the solution for 10 minutes. Cool and filter the contents of the beaker with a little sodium hydroxide solution. Drain the crucible with suction and wash the residue on the filter successively with hot water, dilute acetic acid and finally water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity). Finally, drain the crucible with suction, dry the crucible and residue at $105 \pm 3^\circ\text{C}$ to constant mass, cool in a desiccator and weigh them.

8.2.3 Transfer the residue by means of forceps to the conical flask, add 100 ml of 18 percent (m/m) hydrochloric acid solution per gram of specimen, insert the stopper, shake the flask to wet out the specimen and allow the flask to stand for 15 minutes at room temperature, shaking at intervals. Filter the contents of the flask through a weighed filter crucible and transfer any residual fibres to the crucible by washing out the flask with a little more hydrochloric acid solution. Drain the crucible with suction and wash the residue on the filter successively with hydrochloric acid solution, hot water, dilute ammonia solution and finally cold water, draining the crucible with suction after each addition. (Do not apply suction until each washing liquor has drained under gravity.) Finally, drain the crucible with suction, dry the crucible and residue at $105 \pm 3^\circ\text{C}$ to constant mass, cool in a desiccator and weigh them.

NOTE — The component (protein, nylon 6 or nylon 6.6) present in larger quantity should be dissolved first.

8.2.4 Repeat the procedure prescribed in 8.2.1 to 8.2.3 with the remaining test specimen(s).

8.2.5 Alternatively, using the procedure described above, first dissolve the nylon with hydrochloric acid and subsequently dissolve the protein fibre with sodium hydroxide or potassium hydroxide solution.

8.3 Calculations

8.3.1 Calculate the mass of each component as a percentage of the total mass of the fibres in the mixture by any of the methods prescribed in 7.3.2, 7.3.3, or 7.3.4.

9 REPORT

9.1 The report shall include the following information:

- a) Type of material tested,
- b) Method used (*see* 7 or 8),
- c) Percentage of component fibres in the mixture (individual and average), and
- d) The method used for calculation of percentage of component fibres (*see* 7.3.1 and 8.3.1).

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